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United States Department of Agriculture,

BUREAU OF CHEMISTRY.

OFFICIAL METHOD FOR ANALYSIS OF TANNING MATERIALS.

[Adopted at the Eighteenth Convention of the Association of Official Agricultural Chemists.]

I.—PREPARATION OF SAMPLE.

Barks, woods, leaves, dry extracts, and similar tanning materials should be ground to such a degree of fineness that they can be thoroughly extracted. Fluid extracts must be heated to 50° C., well shaken, and allowed to cool to room temperature.

II.—QUANTITY OF MATERIAL.

In the case of bark and similar material, use such quantity as will give about 0.35–0.45 gram tannins per 100 c. c. of solution, extract in Soxhlet or similar apparatus at steam heat for non-starchy materials. For canaigre and substances containing like amounts of starch use temperature of 50° to 55° C. until near complete extraction, finishing the operation at steam heat. In the case of extract weigh such quantity as will give 0.35–0.45 gram tannins per 100 c. c. of solution, dissolve in 900 c. c. of water at 80°, let stand twelve hours, and make up to 1,000 c. c.

III.—MOISTURE.

(a) Place 2 grams, if it be an extract, in a flat-bottom dish not less than 6 cm. in diameter, add 25 c. c. of water, warm slowly till dissolved, continue evaporation, and dry.

(b) All dryings called for, after evaporation to dryness on water bath or others, shall be done by one of the following methods, the soluble solids and non-tannins being dried under similar and, so far as possible, identical conditions:

1. For eight hours at the temperature of boiling water in a steam bath.
2. For six hours at 100° C, in an air bath.
3. To constant weight in vacuo at 70° C.

IV.—TOTAL SOLIDS.

Shake the solution, and without filtering immediately measure out 100 c. c. with a pipette, evaporate in a weighed dish, and dry to constant weight, at the temperature of boiling water. Dishes should be flat bottom and not less than 6 cm. in diameter.

V.—SOLUBLE SOLIDS.

Double pleated filter paper (S. and S., No. 590, 15 cm.) shall be used. To 2 grams of kaolin add 75 c. c. of the tanning solution, stir, let stand fifteen minutes, and decant as much as possible. Add 75 c. c. more of the solution, pour on filter, keep filter full, reject the first 150 c. c. of filtrate, evaporate the next 100 c. c. and dry. Evaporation during filtration must be guarded against.

VI.—NON-TANNINS.

Prepare 20 grams of hide powder by digesting twenty-four hours with 500 c. c. of water and adding 0.6 gram chrome alum in solution, this solution to be added as follows: One-half at the beginning and the other half at least six hours before the end of the digestion. Wash by squeezing through linen, continue the washing until the wash water does not give a precipitate with barium chlorid. Squeeze thoroughly by hand, and remove as much water as possible by means of

a press, weigh the pressed hide, and take approximately one-fourth of it for moisture determination. Weigh this fourth carefully and dry to constant weight. Weigh the remaining three-fourths carefully and add them to 200 c. c. of the original solution; shake ten minutes, and squeeze the tanned hide through linen. Collect this filtrate, add 5 grams of kaolin, free from soluble salts, stir well and filter through folded filter (S. and S. No. 590, 15 cm.), returning the first 25 c. c. Evaporate 100 c. c. of the clear filtrate. The weight of this residue must be corrected for the dilution caused by the water contained in the pressed hide powder. The shaking must be done in some form of mechanical shaker. The simple machine used by druggists, and known as the milk shake, is recommended.

PROVISIONAL METHOD.

To 14 grams of dry chromed hide powder in a shaker glass add 200 c. c. of the tanning solution, let stand two hours stirring frequently, shake fifteen minutes, throw on funnel with a cotton plug in the stem, let drain, tamp down the hide powder in the funnel, return the filtrate until clear and evaporate 100 c. c.

VII.—TANNINS.

The amount of these is shown by the difference between the soluble solids and the corrected non-tannins.

VIII.—TESTING HIDE POWDER.

(a) Shake 10 grams of hide powder with 250 c. c. of water for five minutes, strain through linen, squeeze the magma thoroughly by hand; repeat this operation three times, pass the last filtrate through paper (S. and S. No. 590, 15 cm.) till clear, evaporate 100 c. c., and dry. If this residue amounts to more than 10 mg. the hide must be rejected.

(b) Prepare a solution of pure gallo-tannin by dissolving 6 grams in 1,000 c. c. of water. Determine the total solids by evaporating 100 c. c. of this solution and drying to constant weight. Treat 200 c. c. of the solution with hide powder exactly as described in paragraph 6. The hide powder must absorb at least 95 per cent of the total solids present. The gallo-tannin used must be completely soluble in water, alcohol, acetone, and acetic ether, and should not contain more than 1 per cent of substances not removed by digesting with excess of yellow mercuric oxid on steam bath for two hours.

IX.—TESTING NON-TANNIN FILTRATE.

(a) *For tannin.*—Test a small portion of the clear non-tannin filtrate with a few drops of a 1 per cent solution of Nelson's gelatin. A cloudiness indicates the presence of tannin, in which case repeat the process described under 6, using 25 instead of 20 grams of hide powder.

(b) *For soluble hide.*—To a small portion of the clear non-tannin filtrate add a few drops of the filtered tannin solution. A cloudiness indicates the presence of soluble hide, in which case repeat the process described under 6, giving the hide powder a more thorough washing.

The temperature of solutions shall be between 16° and 20° when measured or filtered. All dryings should be made in flat-bottom dishes of at least 6 cm. diameter. S. and S. No. 590, 15 cm. filter paper should be used in all filtrations.

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Approved:

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